भारतीय मानक
Indian Standard

IS 17403 : 2020

विस्फोटक और आतिशबाज़ी संयोजन के लिए मैग्नेलियम — विशिष्ट

Magnalium for Explosives and Pyrotechnic Composition — Specification

ICS 71.060.20; 71.100.30

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Explosives and Pyrotechnics Sectional Committee, CHD 26

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Magnalium is an aluminium alloy with magnesium and also contains small amounts of nickel and tin. Alloys with small amounts of magnesium (about 5 percent) have properties of greater strength, greater corrosion resistance and lower density than pure aluminium. Such alloys are used in aircraft and automobile industry. However, alloys with about 50 percent magnesium are brittle and corrode easily. These alloys are flammable when powdered and therefore used in explosives and pyrotechnics. This standard covers the specification of magnalium powder, which is a 50-50 alloy of magnesium and aluminium and is extensively used in the manufacture of explosives and pyrotechnics.

There is no ISO standard on this subject.

The composition of the Committee responsible for formulation of this standard is given in Annex P.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).' The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MAGNALIUM FOR EXPLOSIVES AND PYROTECHNIC COMPOSITION — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and methods of test for magnalium powder (magnesium-aluminium 50 : 50 alloy powder) for use in explosive and pyrotechnic composition.

2 REFERENCES

The standards listed below contain provisions which though reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standards listed below:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*)
4905 : 2015	Random sampling and randomization procedures (*first revision*)
10081 : 1981	Terms relating to commercial explosives, pyrotechnics and blasting practices

3 TERMINOLOGY

For the purpose of this standard, the terms and definitions given in IS 10081 shall apply.

4 REQUIREMENTS

4.1 The material shall consist of an alloy of magnesium and aluminium in approximately equal proportions. It shall be in the form of a clean, bright, free flowing powder without inclusion of any foreign matter.

4.2 The material shall conform to the requirements given in Table 1.

4.3 The material shall satisfy the requirements prescribed in Table 2, when tested as per the method prescribed in Annex M.

NOTES

1 For particulars of IS Sieves, *see* IS 460 (Part 1).

2 Requirements may also be as agreed to between the purchaser and the supplier.

5 PACKING

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in plain steel drums with expandable lids. The drums shall be robust and clean. The interior of the drums shall be free from residual flux and other substances which might react with the material in any manner.

Table 1 Requirements for Magnalium Powder

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, ref to Annex
(1)	(2)	(3)	(4)
i)	Moisture content, percent by mass, *Max*	0.05	A
ii)	Gritty matter, retained on 63 µm IS Sieve, percent by mass, *Max*	0.10	B
iii)	Silicon / compounds of silicon calculated as silicon, percent by mass, *Max*	0.25	C or N
iv)	Iron / compounds of iron calculated as iron, percent by mass, *Max*	0.50	D or N
v)	Zinc / compounds of zinc calculated as zinc, percent by mass, *Max*	0.10	E or N
vi)	Calcium / compounds of calcium calculated as calcium, percent by mass, *Max*	0.10	F or N
vii)	Chlorides calculated as sodium chloride, percent by mass, *Max*	0.03	G
viii)	Other metallic impurities calculated as metals, percent by mass, *Max*	0.50	H or N
ix)	Aluminium / compounds of aluminium calculated as aluminium, percent by mass	48.0 - 52.0	J or N
x)	Magnesium / compounds of magnesium calculated as magnesium, percent by mass	48.0 - 52.0	K or N
xi)	Total metallic magnesium and aluminium, percent by mass, *Min*	96.0	L

Table 2 Requirements after Sieving

(*Clause* 4.3)

Sl No.	Sieve	Requirement
(1)	(2)	(3)
i)	Size 1700 / 300 µm IS Sieve:	
	a) Retained on 1700 µm IS Sieve, percent by mass, *Max*	3
	b) Passing through 300 µm IS Sieve, percent by mass, *Max*	10
ii)	Size 300 / 75 µm IS Sieve:	
	a) Retained on 300 µm IS Sieve, percent by mass, *Max*	3
	b) Passing through 75 µm IS Sieve, percent by mass, *Max*	25
iii)	Size 150 / 75 µm IS Sieve:	
	a) Retained on 150 µm IS Sieve, percent by mass, *Max*	2
	b) Passing through 75 µm IS Sieve, percent by mass, *Max*	45 - 65
iv)	Size 125 / 63 µm IS Sieve:	
	a) Retained on 125 µm IS Sieve, percent by mass, *Max*	2
	b) Passing through 63 µm IS Sieve, percent by mass, *Min*	50
v)	Size 75 µm IS Sieve:	
	Retained on 75 µm IS Sieve, percent by mass, *Max*	3

Alternatively, dry, robust and clean packages containing approved quantity of material, as agreed to between the purchaser and the supplier, may also be used.

6 MARKING

6.1 The drums / packages shall be indelibly and legibly marked with the following details:

a) Name and grade of the material;
b) Manufacturer's name, address and / or his recognized trade mark, if any;
c) Identification in code or otherwise to enable the lot of manufacture to be traced from records;
d) Tare and net mass of the material in the drum;
e) Year and month of manufacture; and
f) Symbol indicating the flammable nature of the material.

6.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 SAMPLING

7.1 General

In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

7.1.1 Samples shall not be taken in an exposed place.

7.1.2 The sampling instrument shall be clean and dry.

7.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

7.1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possibly by suitable means.

7.1.5 The samples shall be placed in clean, dry, airtight glass or other suitable containers which have no action on the material.

7.1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

7.1.7 Each container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* **6**) and the date of sampling.

7.1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

7.2 Scale of Sampling

7.2.1 *Lot*

All containers in a single consignment of the material of the same grade and belonging to the same batch of manufacture shall constitute a lot.

7.2.2 For ascertaining the conformity of material to the requirements of the standard, samples shall be tested from each lot separately.

7.2.3 The number of containers to be selected shall depend on the size of the lot and shall be in accordance with Table 3. These containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 maybe followed.

7.3 Test Samples and Referee Samples

7.3.1 From each of the containers selected according to **7.2.3** draw portions of material from different parts of each container. The quantity of material so drawn shall be sufficient for triplicate determination of all characteristics given in **4**.

7.3.2 Thoroughly mix portions of material drawn from each container. Out of each of these portions, approximately equal quantity of material shall be taken and mixed thoroughly to form a composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample. Each of these parts of composite sample shall be immediately transferred to thoroughly dried bottles which are sealed air-tight with stoppers. These shall be labelled with all the particulars of sample given under **6**.

7.3.3 The referee sample bearing the seals of purchaser and supplier shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

7.4 Number of Tests

All characteristics given in **4** shall be tested on the composite sample.

7.5 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the standard, if all the test results on the composite sample meet the corresponding requirements given in **4**.

Table 3 Scale of sampling

(*Clause* 7.2.3)

Sl No. (1)	Lot Size (2)	No. of Containers to be Selected (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 150	6
v)	151 to 300	7
vi)	301 to 500	8
vii)	501 to 1 000	9
viii)	1 001 and above	10

ANNEX A

[*Table* 1, Sl No. (i)]

DETERMINATION OF MOISTURE

A-1 APPARATUS

A-1.1 Analytical Balance, 0.1 mg accuracy.

A-1.2 Weighing Bottle, squat form, wide mouthed with ground glass stopper (size: 60 mm dia × 40 mm height).

A-1.3 Air Oven, maintained at 105 ± 2°C.

A-1.4 Desiccator, with silica gel as desiccant.

A-1.5 Vacuum Desiccators, filled with concentrated sulphuric acid as desiccant.

A-1.6 Vacuum Pump

A-2 PROCEDURE

Dry a clean weighing bottle with the stopper removed in a hot air oven maintained at 105 ± 2 °C for 1 h. Allow to cool in a desiccator. Insert the stopper and weigh it accurately nearest to 0.1 mg (W_1). Take 5 g of the sample in the weighing bottle, insert the stopper and weigh to the nearest 0.1 mg (W_2). Keep it (half-opened) in the vacuum desiccator filled with concentrated sulphuric acid as desiccant and evacuate the air by vacuum pump and allow the sample to remain in the evacuated desiccator for 24 h. Release the vacuum slowly by allowing dry air to pass in. Cover the stopper and weigh to the nearest 0.1 mg (W_3).

A-3 CALCULATION

$$\text{Moisture content, percent by mass} = \frac{(W_2 - W_3)}{(W_2 - W_1)} \times 100$$

Where,

W_1 = weight of weighing bottle + stopper,

W_2 = weight of weighing bottle + stopper, with sample before desiccation, and

W_3 = weight of weighing bottle + stopper, with the sample after desiccation.

ANNEX B

[*Table* 1, Sl No. (ii)]

DETERMINATION OF GRITTY MATTER

B-1 APPARATUS

B-1.1 Beaker, 500 ml.

B-1.2 Silica Crucible, 30 ml.

B-1.3 Sieve, 63 µm IS sieve.

B-1.4 Small Camel Brush

B-1.5 Whatmann Filter Paper, No. 40, 125 mm dia.

B-2 REAGENTS

B-2.1 Dilute Hydrochloric Acid, 1 : 1 (*v/v*).

B-2.2 Aqua Regia.

B-2.3 Sodium Hydroxide Solution, 50 percent (*m/v*).

B-2.4 Hydrochloric Acid Solution, 2 percent (*v/v*).

B-3 PROCEDURE

Take 5 g of accurately weighed sample in a 500 ml beaker. Add 1 : 1 hydrochloric acid in small portions to dissolve the sample, taking sufficient precautions to avoid any vigorous reaction. When effervescence ceases and the solution becomes clear, decant off the supernatant liquid carefully without losing any residue remaining at the bottom of the beaker. Boil the residue with 25 ml of aqua regia for 15 min. Dilute the acid solution to twice its volume with water. Again decant off the supernatant liquid carefully without losing any residue remaining at the bottom of the beaker. Boil the residue for about 10 min with 20 ml of 50 percent Sodium hydroxide solution. Then dilute immediately with 200 to 300 ml of water. Wash the gritty matter three times with water by decantation and then once with 2 percent hydrochloric acid solution. Filter on a No.40 Whatmann filter paper. Wash and ignite in a silica crucible and then cool. Transfer the ignited gritty matter on to a 63 µm IS sieve and brush it on the surface of the sieve using a small camel brush. After the sieving is complete, weigh the portion retained on the sieve.

B-4 CALCULATION

$$\text{Gritty matter, percent by mass} = \frac{W_1}{W_2} \times 100$$

where

W_1 = mass of grit retained on 63 µm IS Sieve, in g; and

W_2 = mass of the sample taken for test, in g.

ANNEX C

[*Table* 1, Sl No. (iii)]

DETERMINATION OF SILICON CONTENT

C-1 APPARATUS

C-1.1 Beaker, 100 ml, 150 ml and 500 ml.

C-1.2 Silica Crucible, 30 ml.

C-1.3 Tall Neck Beaker, 400 ml.

C-1.4 Analytical Balance, 0.1 mg accuracy.

C-1.5 Whatmann Filters, No. 40, 41 and 42, 125 mm diameter.

C-2 REAGENTS

C-2.1 Concentrated Hydrochloric Acid.

C-2.2 Dilute Sulphuric Acid, 12 ml of concentrated sulphuric acid diluted to 30 ml with water.

C-2.3 Sulpuric Acid Solution, 2 percent (*v/v*).

C-2.4 Ammonium Acetate Solution, 20 percent (*w/v*).

C-2.5 Aqua Regia.

C-2.6 Sodium Hydroxide Solution, 50 percent (*w/v*).

C-2.7 Hydrochloric Acid Solution, 2 percent (*v/v*).

C-3 PROCEDURE

C-3.1 Take 2 g of accurately weighed sample in a 500 ml beaker. Add 50 ml of water. Add 5 ml of concentrated hydrochloric acid. When the first violent reaction of gas has subsided, add further portions of hydrochloric acid up to a total of 25 ml. When the entire sample is dissolved, add dilute sulphuric acid and evaporate the liquid on a sand tray or hot plate until copious fumes are evolved. Cool the residue. Dilute with 200 ml of water. Warm with occasional stirring until the white solid dissolves. Allow the contents of beaker to stand overnight. Filter-off the insoluble residue next morning on a No. 42 Whatmann filter paper. Wash with 2 percent sulphuric acid solution several times (retain the filtrate for the determination of iron and zinc contents as described in Annex D and Annex E).

C-3.2 Extract the residue insoluble in 2 percent sulphuric acid in a 100 ml beaker with 20 ml of hot 20 percent ammonium acetate solution. After 30 min extraction, filter the liquid through a No.41 Whatmann filter paper. Wash the residue once with 20 percent ammonium acetate solution. Dry the residue on the filter paper and ignite in a silica crucible and weigh (M_1).

C-3.3 Warm the ignited residue with 10 ml of aqua regia for 5 min, dilute the solution and filter it through a No.40 Whatmann filter paper. Wash the residue with water, dry and ignite. Transfer the ash to a 150 ml beaker. Add 20 ml of 50 percent of sodium hydroxide solution and boil for 10 min. Wash out the solution and residue into a tall 400 ml beaker containing 200 ml of water. Allow the solid matter to settle for 10 min. Decant off the clear liquid and add a further 200 ml of water.

C-3.4 Repeat the washing by decantation once more and the filter off the residue on a No.40 Whatmann filter paper. Wash the residue on the filter paper with hot water and finally with a 2 percent hydrochloric acid solution. Dry the residue, ignite in a silica crucible and weigh the ash (M_2). The loss in mass (M_1 - M_2) is due to silica derived from elementary silicon originally present as such.

C-4 CALCULATION

$$\text{Silicon, percent by mass} = \frac{(M_1 - M_2) \times 28.08 \times 100}{60.08 \times M_3}$$

Where,

M_1 = mass of the ignited residue remaining after ammonium acetate extraction, in g;

M_2 = mass of the ignited residue remaining after acid and alkali treatment, in g; and

M_3 = mass of sample taken for test, in g.

ANNEX D

[*Table* 1, Sl No. (iv)]

DETERMINATION OF IRON CONTENT

D-1 APPARATUS

D-1.1 Burette, 50 ml.

D-1.2 Pipette, 25 ml.

D-1.3 Conical Flask, 250 ml.

D-1.4 Whatmann Filter Paper, No.4, 125 mm diameter.

D-2 REAGENTS

D-2.1 Concentrated Nitric Acid

D-2.2 Sodium Hydroxide Solution, 30 percent (*v/v*).

D-2.3 Dilute Hydrochloric Acid, 1 : 1 (*v/v*).

D-2.4 Stannous Chloride Solution — Dissolve 6 g of $SnCl_2.2H_2O$ in 6 ml of concentrated hydrochloric acid and dilute to 100 ml.

D-2.5 Mercuric Chloride Solution, saturated.

D-2.6 Phosphoric Acid.

D-2.7 Indicator — Dissolve 0.1 g diphenylamine in 100 ml of concentrated sulphuric acid.

D-2.8 Standard 0.1 N Potassium Dichromate Solution, 4.9 g of $K_2Cr_2O_7$ / 1 000 ml.

D-3 PROCEDURE

D-3.1 To the filtrate retained for iron and zinc (*see* **C-3.1**), add 3 ml of concentrated nitric acid. Boil for about 2 min. Allow the liquid to cool a little and then dilute the solution to about 300 ml. While the solution is still warm, cautiously add 30 percent sodium hydroxide solution with continuous stirring until all aluminium hydroxide first precipitated is just re-dissolved (about 60 ml is required). Warm the solution on the water bath until the brown precipitate of ferric hydroxide has settled. Filter off the $Fe(OH)_3$ precipitate on a No. 4 Whatmann filter paper and wash the precipitate once with hot water. Dissolve the precipitate in about 30 ml of warm 1:1 hydrochloric acid and wash with water. Collect this solution in a 250 ml conical flask. Heat the solution to boil and in this hot condition, add stannous chloride solution drop by drop until the yellow colour due to ferric ions just disappears. Allow to cool.

D-3.2 After cooling to room temperature, add 5 ml of mercuric chloride solution in order to oxidize the excess of stannous chloride. Allow the liquid to stand for a few min and add 5 ml of syrupy phosphoric acid. Add 2 or 3 drops of diphenylamine indicator. Titrate with 0.1 N potassium dichromate solution. The end point is indicated by the appearance of a deep blue colour. Carry out a blank titration using the same quantities of all reagents and apply necessary corrections if any.

NOTE — Preserve the filtrate obtained during filtration of $Fe(OH)_3$ precipitate for the determination of zinc as described in Annex E.

D-4 CALCULATION

$$\text{Percent of Iron, by mass} = \frac{V \times N \times 0.558\ 5 \times 100}{M}$$

Where,

V = volume of standard potassium dichromate solution used in the titration, in ml;

N = normality of standard potassium dichromate solution; and

M = mass of the sample taken for test, in g (*see* **C-3.1**).

ANNEX E

[*Table* 1, Sl No. (v)]

DETERMINATION OF ZINC CONTENT

E-1 APPARATUS

E-1.1 Kipp's Apparatus, to produce hydrogen sulphide gas from ferrous sulphide and hydrochloric acid.

E-1.2 Beaker, 150 ml.

E-1.3 Desiccator

E-1.4 Silica Crucible, 30 ml capacity.

E-1.5 Whatmann Filter Paper, No. 40 and 41, 125 mm diameter.

E-2 REAGENTS

E-2.1 Dilute Hydrochloric Acid, 1 : 1 (*v/v*).

E-2.2 Ammonium Chloride

E-2.3 Dilute Ammonium Hydroxide Solution, 1 : 4 (*v/v*).

E-2.4 Acetic Acid

E-2.5 Ammonium Nitrate Solution, Saturated.

E-2.6 Hydrogen Sulphide Solution, hydrogen sulphide gas saturated in water.

E-3 PROCEDURE

To the filtrate retained for zinc (*see* **D-3.2**), pass hydrogen sulphide gas slowly for 15 min. Keep the liquid steady and undisturbed for 2 h in order to allow any precipitate to settle. Filter the precipitate on a No. 40 Whatmann filter paper. Wash once with hydrogen sulphide solution.

Dissolve the precipitate in 20 ml of warm 1 : 1 dilute hydrochloric acid and wash with water. Collect the dissolution and washings in to a 150 ml beaker. Boil the solution gently and reprecipitate by the addition of ammonium chloride and dilute ammonium hydroxide to the solution until it is just alkaline to litmus paper. Boil for a minute and filter off any precipitate of aluminium hydroxide on a No.41 Whatmann filter paper.

After washing twice with hot water, discard the residue on the filter paper and make the filtrate just acidic with acetic acid. Saturate the solution with hydrogen sulphide, allow the precipitate to settle and filter it off on a No. 40 Whatmann filter paper. Wash the residue once with water which has been previously saturated with hydrogen sulphide gas. Moisten the paper with a few drops of ammonium nitrate solution. Place it in a silica crucible. Dry, ignite, cool in a desiccator and weigh (the colour of the ash, yellow while hot, confirms the presence of zinc).

Calculate the zinc equivalent from the mass of the ignited precipitate as ZnO and express it as the percentage of the mass of original sample.

E-4 CALCULATION

$$\text{Zinc, percent by mass} = \frac{M_1 \times 65.38 \times 100}{81.38 \times M_2}$$

Where,

M_1 = mass of ignited residue, in g; and

M_2 = mass of the sample taken for test (*see* Annex C), in g.

ANNEX F

[*Table* 1, Sl No. (vi)]

DETERMINATION OF CALCIUM

F-1 APPARATUS

F-1.1 Beaker, 250 ml and 500 ml.

F-1.2 Water Bath

F-1.3 Hot Plate

F-1.4 Asbestos Padded Gooch Crucible

F-1.5 Whatmann Filter Paper, No. 4, 125 mm diameter.

F-2 REAGENTS

F-2.1 Concentrated Hydrochloric Acid

F-2.2 Dilute Sulphuric Acid, 1 : 6 (*v/v*).

F-2.3 Sodium Sulphate, free from calcium.

F-2.4 Denatured Spirit

F-2.5 Dilute Ammonium Hydroxide Solution, 1 : 4 (*v/v*).

F-2.6 Dilute Acetic Acid, 6 percent (*m/v*).

F-2.7 Ammonium Oxalate Saturated Solution

F-2.8 Standard 0.1 N Potassium Permanganate Solution

F-3 PROCEDURE

F-3.1 Take 5 g of accurately weighed sample in a 500 ml beaker. Add 50 ml of water. Dissolve the sample by adding in small portions of concentrated hydrochloric acid, not exceeding 50 ml or till the sample dissolves completely, whichever is earlier. Add 2 g of sodium sulphate. Evaporate the solution on a water bath just to a point when a solid layer begins to form on the surface of the liquid. Remove the beaker from the water bath and add 400 ml of denatured spirit.

F-3.2 Stir the liquid and allow it to stand overnight. Filter through a No. 4 Whatmann filter paper and wash the residual salts with denatured spirit to remove the magnesium chloride. Place the filter paper containing the residual salts in the original beaker and warm over hot plate to remove denatured spirit. Dissolve the residual salts in 50 ml of hot water containing a few ml of concentrated hydrochloric acid. Filter into a 250 ml beaker. After neutralization with ammonium hydroxide solution, make the solution just acidic with dilute acetic acid.

F-3.3 Add 25 ml of cold saturated ammonium oxalate solution. Boil the solution for 10 min. Allow to stand for about 2 h. Filter off the precipitate formed on an asbestos padded Gooch crucible. Wash with cold water until free from soluble oxalate. Transfer the precipitate to a beaker containing 50 ml of hot dilute sulphuric acid. Stir and titrate with standard potassium permanganate solution till the light pink colour of permanganate persists.

F-4 CALCULATION

$$\text{Calcium, percent by mass} = \frac{V \times N \times 0.02 \times 100}{M}$$

Where,

V = volume of standard potassium permanganate solution;

N = normality of standard potassium permanganate solution; and

M = mass of sample taken for test, in g.

ANNEX G

[*Table* 1, Sl No. (vii)]

DETERMINATION OF CHLORIDE

G-1 APPARATUS

G-1.1 Beaker, 250 ml

G-1.2 Conical Flask, 250 ml

G-1.3 Burette

G-1.4 Whatmann Filter Paper, No. 4, 125 mm diameter

G-2 REAGENTS

G-2.1 Potassium Chromate Solution, 5 percent (*m*/*v*)

G-2.2 Calcium Carbonate

G-2.3 Standard Silver Nitrate Solution, 0.1 N.

G-3 PROCEDURE

Take 5 g of accurately weighed sample in a 250 ml beaker. Add 50 ml of water. Warm gently, and then cool. Filter through a No. 4 Whatmann filter paper. Wash with water. Collect the filtrate and washings in a 250 ml conical flask. Add 0.1 g of calcium carbonate and thoroughly mix. Add few drops of 5 percent potassium chromate solution. Titrate with standard 0.1 N silver nitrate solution until to get the reddish tinge. Carry out the blank test without sample.

G-4 CALCULATION

Chloride as NaCl, percent by mass =

$$\frac{(V - v) \times N \times 0.058\ 45 \times 100}{M}$$

Where,

V = volume of standard silver nitrate solution consumed for the sample;

v = volume of standard silver nitrate solution consumed for the blank;

N = normality of standard silver nitrate solution; and

M = mass of sample taken for test, in g.

ANNEX H

[*Table* 1, Sl No. (viii)]

DETERMINATION OF OTHER METALLIC IMPURITIES

H-1 APPARATUS

H-1.1 Kipp's Apparatus, to produce hydrogen sulphide gas from ferrous sulphide and hydrochloric acid.

H-1.2 Beaker, 500 ml.

H-1.3 Silica Crucible, 30 ml capacity.

H-1.4 Desiccator

H-1.5 Whatmann Filter Paper, No. 40, 125 mm diameter.

H-2 REAGENTS

H-2.1 Concentrated Hydrochloric Acid

H-2.2 Concentrated Nitric Acid

H-2.3 Dilute Nitric Acid, 1 : 1 (*v*/*v*)

H-2.4 Concentrated Sulphuric Acid

H-2.5 Bromine Water, saturated solution

H-2.6 Ammonium Chloride

H-2.7 Dilute Ammonium Hydroxide Solution, 1 : 4 (*v*/*v*)

H-2.8 Hydrogen Sulphide Solution, hydrogen sulphide gas saturated in water

H-3 PROCEDURE

Take 5 g of accurately weighed sample in a 500 ml beaker. Add 50 ml of water. Add 10 ml of concentrated Hydrochloric acid. Warm the liquid initially until the reaction commences. When the first violent reaction of gas has subsided, add further portions of hydrochloric acid slowly as required to dissolve the sample completely, but not more than 60 ml. When the entire sample has dissolved, add 10 ml of concentrated nitric acid and gently boil for 15 min. Cool and dilute to twice the volume and filter if necessary.

To the filtrate, add 10 g of ammonium chloride and make it just alkaline with dilute ammonium hydroxide solution. Pass hydrogen sulphide gas through the alkaline solution till precipitation is complete. Filter the precipitated sulphides through a No. 40 Whatmann filter paper. Wash the precipitate with water which has been previously saturated with hydrogen sulphide gas. Dissolve the precipitate in dilute nitric acid. Add 5 ml of concentrated sulphuric acid. Evaporate the solution to fuming for 15 min. Cool the solution. Dilute it to exactly 100 ml with water.

Allow to stand overnight. Filter. Through the filtrate pass hydrogen sulphide gas for 15 min. Filter and wash the precipitate with water which has been previously saturated with hydrogen sulphide gas. Dry the precipitate. Ignite it in a tared silica crucible. Cool in a desiccator. Weigh.

H-4 CALCULATION

Other metallic impurities, percent by mass =

$$\frac{M_1 \times 63.546 \times 100}{79.55 \times M_2}$$

Where,

M_1 = mass of the ignited residue, in g; and
M_2 = mass of the sample taken for test, in g.

ANNEX J

[*Table* 1, Sl No. (ix)]

DETERMINATION OF ALUMINIUM

J-1 APPARATUS

J-1.1 Beaker, 500 ml

J-1.2 Volumetric Flask, 500 ml

J-1.3 Pipette, 50 ml

J-1.4 G-3 Sintered Glass Crucible

J-1.5 Desiccator

J-1.6 Whatmann Filter Paper, No. 41, 125 mm diameter

J-2 REAGENTS

J-2.1 Concentrated Hydrochloric Acid

J-2.2 Dilute Ammonium Hydroxide Solution, 1 : 1 (*v/v*).

J-2.3 Glacial Acetic Acid

J-2.4 Ammonium Chloride

J-2.5 Hydroxylamine Hydrochloride, 1 percent (*m/v*).

J-2.6 Ammonium Benzoate Solution — Dissolve 100 g of pure ammonium benzoate in 1 l of warm water and add 1 mg of Thymol as a preservative.

J-2.7 Ammonium Tartarate Solution — Dissolve 30 g of ammonium tartarate in 500 ml of water and 120 ml of ammonium hydroxide solution and dilute to 1 l with water.

J-2.8 Benzoate Wash Solution — To 100 ml of ammonium benzoate solution, add 900 ml of warm water and 20 ml of glacial acetic acid.

J-2.9 8-Hydroxyquinoline (Oxine) Solution — Dissolve 50 g of reagent grade 8-hydroxyquinoline, C_9H_6NOH, in 120 ml of glacial acetic acid and dilute to 1 l with water.

J-3 PROCEDURE

Take 0.4 to 0.5 g of accurately weighed sample in a 500 ml beaker. Add 50 ml of water. Dissolve the sample by adding in small portions of a total of 10 ml of concentrated hydrochloric acid. Complete the dissolution. Cool to room temperature and transfer into 500 ml volumetric flask. Make up to 500 ml with water. Pipette a 50 ml aliquot into a 500 ml beaker and dilute with 50 ml of water. Neutralize the solution with 1 : 1 ammonium hydroxide solution by adding dropwise with continuous stirring until all the free acid is neutralized without permanent precipitation of aluminium hydroxide.

Add 1 ml of glacial acetic acid, 1 g of ammonium chloride and 10 ml of 1 percent hydroxylamine hydrochloride and 20 ml of ammonium benzoate solution. Heat the mixture to boiling with stirring. Keep at gentle boiling for 5 min and then filter on a No. 41 Whatmann filter paper. Wash the precipitate 8 to 10 times with hot benzoate wash solution. Dissolve the precipitate with 10 ml portions of hot ammonium tartrate solution, washing with hot water after portion is added. Collect the solution in the original beaker and dilute to 150 to 200 ml with water.

Heat the solution to 70 to 90 °C. Add 20 ml of 8-hydroxyquinoline (Oxine) solution and digest for 15 min without boiling. Take a tared G-3 sintered glass crucible. Filter the solution through this tared G-3 sintered glass crucible. Wash the crucible thoroughly with hot water, upto 8 times for transferring of all the precipitate. Dry the precipitate for 2 h at 120 to 130 °C. Cool in a desiccator. Weigh the crucible with precipitate, in which the precipitate is as aluminium oxinate, $Al\,(C_9H_6ON)_3$.

J-4 CALCULATION

Aluminium, percent by mass =

$$\frac{(M_3 - M_2) \times 26.98 \times 500 \times 100}{459.25 \times 50 \times M_1}$$

Where,

M_1 = mass, in g, of the sample taken for test;

M_2 = mass, in g, of G-3 sintered glass crucible; and

M_3 = mass, in g, of sintered glass crucible with aluminium oxinate precipitate.

ANNEX K

[*Table* 1, Sl No. (x)]

DETERMINATION OF MAGNESIUM

K-1 APPARATUS

K-1.1 Beaker, 500 ml.

K-1.2 G-3 Sintered Glass Crucible

K-1.3 Electric Bunsen

K-1.4 Muffle Furnace

K-1.5 Desiccator

K-1.6 Whatmann Filter Paper, No. 41, 125 mm diameter.

K-2 REAGENTS

K-2.1 Sodium Hydroxide Solution, 15 percent (*m*/*v*)

K-2.2 Hydrogen Peroxide, 6 percent (*v*/*v*)

K-2.3 Sodium Carbonate Solution, 0.5 percent (*m*/*v*)

K-2.4 Dilute Hydrochloric Acid, 1 : 1 (*v*/*v*)

K-2.5 Dilute Ammonium Hydroxide Solution, 1 : 1 (*v*/*v*)

K-2.6 Dilute Ammonium Hydroxide Solution, 1 : 10 (*v*/*v*)

K-2.7 Dilute Ammonium Hydroxide Solution, 1 : 20 (*v*/*v*)

K-2.8 Diammonium Hydrogen Phosphate Solution, 10 percent (*m*/*v*)

K-3 PROCEDURE

Take 0.1 g of accurately weighed sample in a 500 ml beaker. Dissolve in 15 percent sodium hydroxide solution.

Heat the solution after the initial reaction has subsided. When the hydrogen evolution ceases, add a few drops of 6 percent hydrogen peroxide. Dilute to 150 ml with hot water. Allow the precipitate to settle. Filter through a No. 41 Whatmann filter paper. Wash with hot 0.5 percent sodium carbonate solution. Dissolve the residue on the filter paper by adding 40 ml of 1 : 1 hydrochloric acid and wash with hot water.

Neutralize the filtrate to methyl red with ammonium hydroxide adding a few drops in excess. Evaporate the filtrate to 75 ml. Acidify the filtrate to methyl red with dilute hydrochloric acid. Boil. Add 10 ml of 10 percent diammonium hydrogen phosphate solution and a few drops of 1 : 10 ammonium hydroxide solution with stirring until precipitation is complete followed by addition of an excess of 5 ml of ammonium hydroxide solution. Stir vigorously and allow to stand overnight. Filter on a No. 41 Whatmann filter paper. Wash with dilute 1 : 20 ammonium hydroxide solution. Dry at 100°C for 4 h.

Take a tared Silica crucible. Transfer the paper with precipitate in the tared silica crucible. Ignite initially on a Bunsen burner and finally in a muffle furnace at 1 100 °C for 30 min. Remove the crucible from the muffle furnace. Cool it outside for 5 min and then finally cool to room temperature in a desiccator. Weigh the crucible with the ignited mass of precipitate, in which the ignited mass of precipitate is as magnesium pyro phosphate, $Mg_2P_2O_7$.

K-4 CALCULATION

Magnesium, percent by mass =

$$\frac{(M_3 - M_2) \times 48.624 \times 100}{222.572 \times 50 \times M_1}$$

where,

M_1 = mass of the sample taken for test, in g;
M_2 = mass of silica crucible, in g; and
M_3 = mass of silica crucible with the ignited mass as magnesium pyro phosphate, in g.

ANNEX L

[*Table* 1, Sl No. (xi)]

DETERMINATION OF TOTAL METALLIC MAGNESIUM AND ALUMINIUM

L-1 The percentage of metallic magnesium and metallic aluminium, as determined by measurement of the H_2 gas liberated when the alloy is treated with dilute H_2SO_4 and assuming the free metals to be present in the same ratio as the total percentage of magnesium and aluminium, together shall not be less than 96 percent.

L-2 APPARATUS

The apparatus shall consist of the following main parts assembled as shown in Fig.1.

L-2.1 Lunge Nitrometer, provided with water jacket.

L-2.2 Reaction Bottle, capacity of about 60 ml.

L-2.3 Glass Weighing Tube, closed at one end, about 40 mm long and having an internal diameter of 5 mm.

L-3 REAGENTS

L-3.1 Dilute Sulphuric Acid, 10 percent (*v/v*);

L-3.2 Methyl Red Solution — Dissolve 0.3 g of methyl red in water and dilute to 1 000 ml with water.

L-4 PROCEDURE

Weigh accurately 0.1 g of sample in to the weighing tube. Introduce the tube containing the sample into the reaction bottle. Add carefully 20 ml of 10 percent dilute sulphuric acid through a funnel to the reaction bottle and connect it to the lunge nitrometer filled with acidulated water, and ensure air tightness of system. Make sure that the air in the reaction bottle is initially at atmospheric pressure.

Expel all the air completely from the nitrometer bulb by bringing up the level of acidulated water coloured with a drop of methyl red solution. Note the temperature of the water jacket around the nitrometer and of the water bath around the reaction bottle, which should be almost the same.

Tilt the reaction bottle in such a way that the sulphuric acid finds its way into the weighing tube containing the sample, at the same time opening the passage to the nitrometer. When the reaction ceases, adjust the pressure very nearly to the atmospheric pressure and allow the apparatus to stand for 1 h. Add a drop or two of methyl red solution in the cup of the nitrometer to serve as an indicator of the pressure of the gas in the nitrometer through water level.

Find out by trial, the position at which the liquid in the nitrometer cup does not move up or down the bore of the stopper when the nitrometer is open to the atmosphere. This operation is to be repeated after putting the gas in the nitrometer in communication with that in the reaction bottle, to ensure that the air shut off in the latter is at atmospheric pressure.

After the reaction, read the volume of the gas. Read the temperature of water in the jacket and the water-bath around the reaction bottle. Note the barometric reading and the temperature

L-5 CALCULATION

Total metallic magnesium and aluminium =

$$\frac{(P-p)\times V\times 273\times 100\times 100}{760\times 108\ 340\times (273+t)\times M}$$

Where,

P = atmospheric pressure of mercury at the instant of measurement of volume of hydrogen gas produced, in mm;

p = vapor pressure of mercury of acidulated water at t °C;

V = volume of Hydrogen gas produced, in ml;

t = temperature at the instant of measurement of the volume of Hydrogen gas produced, in °C; and

M = mass of sample taken for the test, in g.

NOTES

1 100 g of pure metallic magnesium will produce 921 00 ml of hydrogen gas, measured at NTP.

2 100 g of pure metallic aluminium will produce 124 580 ml of hydrogen gas, measured at NTP.

3 100 g pure metallic magnesium and aluminium in an alloy of magnalium powder will produce 108 340 ml of hydrogen gas, measured at NTP.

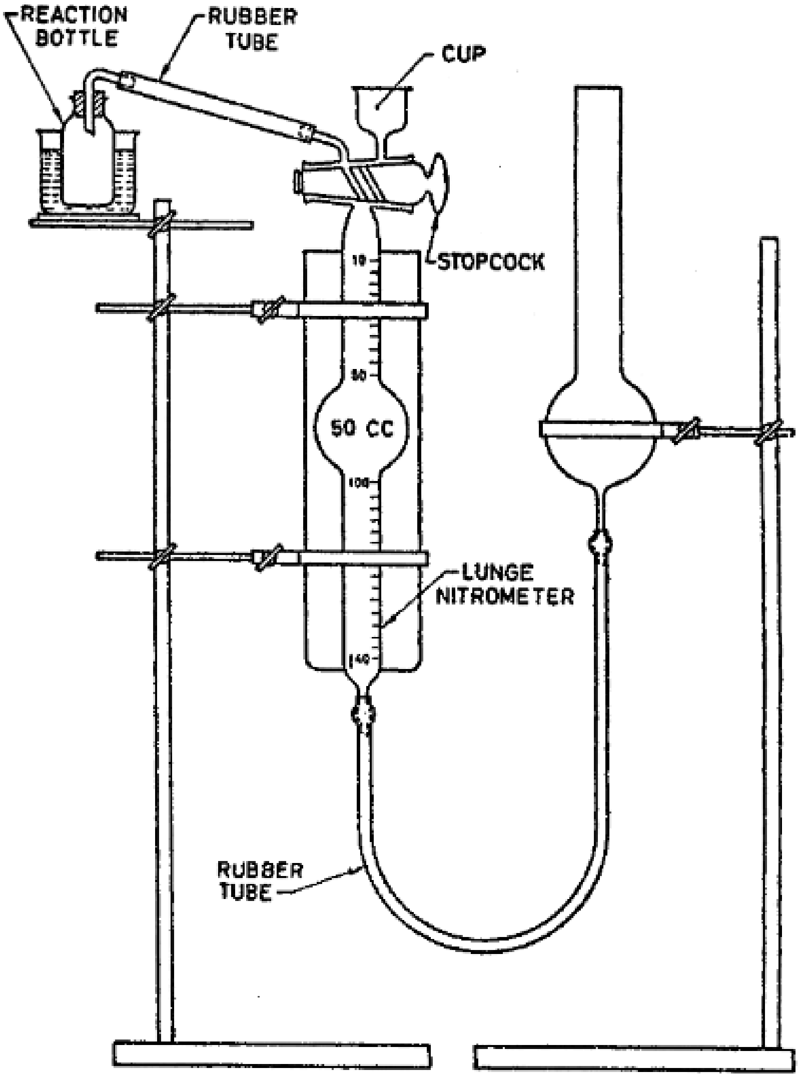

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF TOTAL METALLIC MAGNESIUM AND ALUMINIUM

ANNEX M

[*Table* 1, Sl No. (xii)]

SIEVE ANALYSIS

M-1 Mechanical (rotary-sieve shaker instrument) or manual (hand sieve with brushing) methods may be used for the dry sieve analysis of metal powders.

M-2 SAMPLE CONDITION FOR DRY SIEVE ANALYSIS

M-2.1 Sample of granular metallic powders shall be in dry, unlubricated form.

M-2.2 The powder shall be tested in the as-received condition.

M-2.3 In certain instances, the powder may be dried at 110 ± 5°C for 1 h.

M-2.4 If the powder is susceptible to oxidation, the drying should take place in an inert gas.

M-3 APPARATUS

M-3.1 Test Sieves, of required aperture size (1 700 µm, 300 µm, 150 µm, 125 µm, 75 µm and 63 µm IS Sieves), 8 inches in diameter and 2 inches in depth, brass / SS frame fitted with SS wire woven cloth, 8-inch diameter lid and collecting pan.

M-3.2 Analytical Balance, having 1 mg accuracy.

M-3.3 Soft Brush, 2 inch (50 mm).

M-3.4 Ro-tap Sieve Shaker — Mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 270 – 300 per minute and a tapping action of 140 – 160 per minute. The sieve shaker shall be fitted with a plug to receive the impact of tapping device of 3 kg hammer with its free falling height of 60 mm. The entire apparatus shall be mounted rigidly and preferably shall be provided with a time switch to ensure accuracy of duration of the test.

The instrumental method is not applicable to the powders if the:

a) morphology of powder differs markedly from being equiaxial and the particle size is wholly (or) mostly under 45 µm; and
b) particles tend to be clogged with the sieve surface during ro-tap-sieve.

In the above cases, an alternate manual method shall be followed up.

M-4 PROCEDURE

M-4.1 Mechanical Sieving by Ro-tap Sieve Shaker

The nest of sieves selected shall be assembled in consecutive order based on size of openings. The sieve having the coarsest opening shall be placed at the top and the assembly being completed by a solid collecting pan placed below the bottom sieve.

Take 100 g of sample if its apparent density is greater than 1.5 g/cm^3 (or) take 50 g of sample if its apparent density is less than 1.5 g/cm^3. Transfer 50 g of the sample on the uppermost sieve of the coarsest aperture size and close this sieve with a solid lid.

The sieve assembly shall be fastened securely in the ro-tap sieve shaker. Operate the sieve shaker for 15 min. The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them on a glazed paper.

Any material adhering to the bottom of the sieve and frame shall be brushed with the soft brush into the next finer sieve. The sieve just removed then shall be tapped upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed.

This process shall be repeated for each sieve in the nest, and the fraction collected in pan shall also be removed and weighed.

The sum of the masses of all the fractions shall be recorded.

The difference between the sum and the total sample mass shall be added to the mass of the fraction collected in the bottom pan, provided the sum of all the fractions is not less than 99 percent of the original sample mass (if the sum is less than 99 percent, check for weighing errors, pinholes, etc, and repeat the test).

M-4.2 Manual Sieving By Hand Sieve with Brushing

The nest of sieves selected shall be assembled in consecutive order based on size of openings.

The sieve having the coarsest opening shall be placed at the top and the assembly being completed by a solid collecting pan placed below the bottom sieve. Take 10 g of the sample and place it on the top sieve. Brush it gently using the soft brush. Carry out the brushing for 15 min or until no further material passes through the sieve (whichever is the shortest). Weigh the mass of the material retained on the sieve.

Continue this brushing on each sieve and weighing the retention on each sieve and pan.

Express the mass of material retained by the sieve as the percentage of the original sample.

M-5 CALCULATION

The mass of the fractions retained on each sieve and the mass of the fraction collected in the pan shall be expressed as the percentage of the test specimen mass to the nearest 0.1 percent.

Material retained on each sieve, percent by mass =

$$\frac{M_1 \times 100}{M}$$

Where,

M = mass of test specimen taken, in g; and

M_1 = mass of the fraction retained on sieve, in g.

If mass fraction is less than 0.1 percent, it shall be reported as "trace". If fraction is absent, it shall be reported as '0.0'.

ANNEX N

[*Table* 1, Sl. No. (xiii)]

CHEMICAL ANALYSIS USING ATOMIC ABSORPTION SPECTROPHOTOMETER

N-1 APPARATUS

N-1.1 Atomic Absorption Spectrophotometer — The manufacturer's instruction shall be followed for AAS instrumental analysis.

N-1.2 Autoclave

N-1.3 PVC Volumetric Flask, transparent, 250 ml and 1 000 ml.

N-1.4 Beaker, 250 ml.

N-1.5 Pipette, 25 ml.

N-1.6 Silica Crucible

N-1.7 Muffle Furnace

N-1.8 Desiccator

N-1.9 Whatmann Filter Paper, No.41, 125 mm diameter.

N-2 REAGENTS

N-2.1 Hydrofluoric Acid, 48 percent (*v/v*).

N-2.2 Concentrated Nitric Acid

N-2.3 Dilute Nitric Acid, 1 : 1 (*v/v*).

N-2.4 Concentrated Hydrochloric Acid

N-2.5 Dilute Hydrochloric Acid, 1 : 1 (*v/v*).

N-2.6 High Purity Silica Powder

N-2.7 High Purity Iron Granules

N-2.8 High Purity Zinc Metal

N-2.9 High Purity Calcium Carbonate Powder

N-2.10 High Purity Copper Granules

N-2.11 High Purity Aluminium Metal

N-2.12 High Purity Magnesium Metal/Ribbon

N-3 PREPARATION OF STANDARD STOCK SOLUTIONS

N-3.1 Standard Stock Solution of Silicon

Dissolve 0.536 g of 99.9 percent high pure silica powder with 10 ml of hydrofluoric acid and 10 ml of concentrated nitric acid in autoclave for 1 h at 110 °C. Cool and make up to 250 ml with water in a PVC volumetric flask.

$$\text{Concentration of silicon} = \frac{99.9 \times 0.536 \times 28.08 \times 4000}{100 \times 60.08}$$

Each 1 ml of solution contains 1 mg of silicon.

Make appropriate dilution to prepare the standard solutions of required concentrations in PVC volumetric flasks.

N-3.2 Standard Stock Solution of Iron

Dissolve 1 g of 99.9 percent minimum pure iron granules in 50 ml of 1 : 1 Nitric acid. Boil to expel nitrous fumes and cool. Make up the solution in 1 000 ml volumetric flask with water.

Each 1 ml of solution contains 1 mg of iron. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-3.3 Standard Stock Solution of Zinc

Dissolve 1 g of 99.9 percent minimum pure zinc metal in 50 ml of 1 : 1 hydrochloric acid. Boil gently and cool. Make up the solution in 1 000 ml volumetric flask with 1 percent hydrochloric acid.

Each 1 ml of solution contains 1 mg of zinc. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-3.4 Standard Stock Solution of Calcium

To a 2.5 g of 99.9 percent minimum pure calcium carbonate powder, add 50 ml of water and add dropwise a minimum volume of concentrated hydrochloric acid. Boil to effect the completion of dissolution and cool. Make up the solution in 1 000 ml volumetric flask with water.

Each 1 ml of solution contains 1 mg of calcium. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-3.5 Standard Stock Solution of Copper

Dissolve 1 g of 99.9 percent minimum pure copper granules in 50 ml of 1 : 1 nitric acid. Boil to expel nitrous fumes and cool. Make up the solution in 1 000 ml volumetric flask with water.

Each 1 ml of solution contains 1 mg of copper. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-3.6 Standard Stock Solution of Aluminium

Dissolve 1 g of 99.9 percent minimum pure aluminium metal in 50 ml of 1 : 1 hydrochloric acid, adding a small drop of mercury as a catalyst. Boil and cool. Filter the solution to remove mercury. Wash and make up the solution in 1 000 ml volumetric flask with 1 percent hydrochloric acid.

Each 1 ml of solution contains 1 mg of aluminium. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-3.7 Standard Stock Solution of Magnesium

Dissolve 1 g of 99.9 percent minimum pure magnesium metal/ribbon in minimum volume of 1 : 1 hydrochloric acid. Boil and cool. Make up the solution in 1 000 ml volumetric flask with 1 percent hydrochloric acid.

Each 1 ml of solution contains 1 mg of magnesium. Make appropriate dilution to prepare the standard solutions of required concentrations.

N-4 PROCEDURE

Take 0.5 g of accurately weighed sample in a 250 ml beaker. Add 15 ml of water. Add 15 ml of concentrated hydrochloric acid in small portions to dissolve the sample, taking sufficient precautions to avoid any vigorous reaction. Add 10 ml of 1 : 1 nitric acid. Boil to dissolve and then cool. If insoluble residue is present, filter through a No.41 Whatmann filter paper. Wash with water. The residue collected in the filter paper is dried and ignited for the determination of percentage of acid insoluble matter.

Collect the filtrate and washings in a 250 ml volumetric flask. Make up to 250 ml with water. This solution is fed for the determination of silicon, iron, zinc, calcium, and other metallic impurities as copper.

Dilute the solution to 25 times for the determination of aluminium and 250 times for the determination of magnesium.

Prepare an acid blank.

Determine the element of interest by atomic absorption spectrophotometer using the concerned wavelength as mentioned in Table 4. The concentration of element content is observed in ppm. From this, the percentage of element can be calculated.

Table 4 Wavelengths Corresponding to the Elements of Interest

(*Clause* N-4)

Sl No. (1)	Element (2)	Wavelength (nm) (3)
i)	Silicon	251.6
ii)	Iron	248.3
iii)	Zinc	213.9
iv)	Calcium	422.7
v)	Copper	324.7
vi)	Aluminium	309.3
vii)	Magnesium	285.2

5 CALCULATION

For silicon, iron, zinc, calcium and copper, percentage of element = $\frac{C}{40 \times M}$

Percentage of aluminium = $\frac{C \times 25}{40 \times M}$

Percentage of magnesium = $\frac{C \times 250}{40 \times M}$

Where,

C = concentration of the respective element observed by AAS, in ppm; and

M = mass of sample taken for the test, in mg.

ANNEX P

(*Foreword*)

COMMITTEE COMPOSITION

Explosives and pyrotechnics sectional committee, CHD 26

Organization	*Representative(s)*
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Ayyan Fireworks, Sivakasi	Shri G. Abiruben
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